^{3}He Top Loading Cryostat
User Manual

Contents:

I. Introduction:

1. This is a complicated system that should only be run by an experienced user or member of the sample environment team. This manual is not meant to be a substitute for that, but instead should be a point of reference and a learning tool. For more information contact: Evan Fitzgerald (301-975-6657) or Dan Dender (301-975-6225).
2. What makes this system unique is that it is a *top loading* ^{3}He system. Along with this feature come some inherent problems that had to be overcome to get the system to work. To reach a base temperature of 300mK the sample stick must make solid contact with the ^{3}He pot and, for thermal shielding it, must also make contact with the 1K pot and the ^{4}He reservoir. To accomplish this a force-generator was designed which pushes down on the sample stick and mates matching surfaces on the stick and the inside of the sample tube. The mating points are then connected by copper braids to the respective parts of the system. The system, however, still needs more cooling power to over come the large thermal mass of the stick and assembly, so there are actually two identical, independent ^{3}He systems (insert A and B).

II. Valve Names:

1. V-1A/B = Charcoal cooling control
2. V-2A/B = 1K pot needle valve
3. V-3 = Vacuum space safety pressure relief
4. V-4A/B = ^{3}He dump fill
5. V-5A/B = 1K pot pumping and over pressure
6. V-6A/B = Charcoal cooling flow gauge
7. V-7 = Air lock double o-ring evacuation
8. V-8 = Air lock evacuation

9. V-9 = Sample well gate valve
10. V-10 = Inner vacuum chamber (IVC) evacuation
11. V-11 = IVC safety pressure relief
12. V-12 = Vacuum jacket evacuation
13. V-13 = Helium reservoir pressure relief

III. Sample Loading:

1. With the system at room temperature mount the sample stick on top of the cryostat and bolt into place. Open V9 and slide the stick slowly into the sample well until it is stopped by the 0.5K thermal anchor stage. Use the force generator to compress the stick down approximately 0.3". Do NOT tighten the force generator all the way.
2. After checking that the hose between V7 and V8 is properly connected close both valves and pump out the IVC through V10 with a turbo pump. Once the vacuum reaches less than 5 $x10^{-4}$ Torr disconnect the turbo pump and connect a leak detector to V10.
3. Leak check the stick's hoses, valves, seals and connectors. Using a rough pump evacuate the 1K pot through V-5A/B, leaving V-2A/B closed, then backfill with He gas while monitoring the leak detector. Leave the 1K pot filled with He gas and the IVC evacuated. Disconnect the leak detector.

IV. Liquid Nitrogen Cooldown:

1. Add a small amount of ^{3}He gas into the IVC through V-10 (this should be done by an experienced user because of the cost of ^{3}He gas). There is a setup stored with the ^{3}He gas bottle that has the appropriate connections to make this step a bit easier.

2. Using a rough pump evacuate the helium reservoir through V-13, backfill with He gas and repeat three times, the last time leaving it filled with He gas. Start filling the helium and nitrogen reservoirs with LN_2.
3. Attach the rough pump to V-5A/B and pump on the 1K pot. Throughout the fill occasionally open V-2A/B one turn and then close it. This will keep the needle valve from getting stuck. Stop this procedure when the 1K pot reaches less than 100K or when you stop filling the He reservoir with LN_2.
4. Close V-2A/B and pump out the 1K pot then back fill with He gas and seal off.
5. Gently tighten the force generator if necessary as the stick will shrink as it cools, but do not over-tighten.
6. Let the LN_2 in the nitrogen reservoir fill all the way to the top, but stop filling the helium reservoir after approximately one hour or when the LN_2 level meter reads above 70%.
7. Let the system sit overnight to completely cool to Nitrogen temperature.

V. Liquid Helium Cooldown

1. If by the next morning the sensors read greater than 100K add more nitrogen to the helium reservoir and let things cool completely.
2. If the sensors are all reading near LN_2 temperature then blow the LN_2 out of the helium reservoir by inserting a tube into the fill port and applying 3-5psi with helium gas into V-13. The fill port can slide up and down, so it needs to be pushed all the way down and then screwed in to place. The nitrogen will flow out the tube, so use a rubber hose to direct it into a cryogenic liquid container.

3. When all of the LN_2 is out use a rough pump to evacuate the helium reservoir through V-13, backfill with He gas and repeat three times, the last time leaving it filled with He gas.
4. Start transferring liquid helium (LHe) through the helium fill port, but keep the pressure low (<3psi).
5. Monitor the temperatures of both ^{3}He inserts to make sure they cool steadily. When their temperatures reach approximately 25K start evacuating the IVC through V-10 using a turbo pump and allow to pump overnight (at least a few hours are necessary).
6. Without the exchange gas in the IVC cooling of the sample stick is dependant on the contact to the multiple mating surfaces. Monitor the temperature of the 1K stage on the stick and continue to tighten the force generator until it stops cooling and equilibrates with the 1K stage of the insert. Be careful not to over-tighten the force generator.
7. When the LHe is full, level meter reads 100% and large dense plume of exhaust, remove the transfer line then seal the reservoir. V-13 should be sealed with the over-pressure valve to maintain a pressure of about 3psi in the helium reservoir.

VI. Cooling to Base Temperature:

1. If the helium was recently filled wait a few minutes for some pressure to build up in the reservoir. Open V-1 and V-6 and verify flow by seeing the small black indicator ball jump up to a flow rate. The small black knob on the flow meter may also have to be adjusted, but if the ball doesn't move even with a light tap

close V-1 or V-6 immediately to prevent air from being sucked back in. Close both V-1 and V-6.

2. If you are certain all of the He exchange gas is out of the IVC begin pumping on the 1K pot. Connect a high volume rough pump to V-5A/B and pump out the lines back to V-2A/B. Slowly open V-2A/B and adjust to reach the lowest sustainable base temperature (~1.1 – 1.3 K).
3. When the 1K pots are steadily below 2K set the temperatures of the charcoal adsorption pumps at 20K by turning the heater on the 331 controller (top right) to Medium. The 1K pots will briefly rise while they cool the warm ^{3}He gas. Each time they cool back below 2K raise the set temperature of the charcoal pump 5 more degrees until you reach 40K. The sample temperature should slowly decrease to around 1.8K. The ^{3}He is condensing when the ^{3}He pots reach 1.8K. Continue to condense for at least half an hour after this point (1 hour condensation = ~25hours at base; 4 hours of condensation = ~39hours at base).
4. Once the ^{3}He has condensed for the desired amount of time turn off the charcoal adsorption pump heater and slowly open V-1 and V-6. Once again watch to see the black ball on the flow meter measure a flow rate and close the valve if it doesn't move. The recommended flow rate is approximately 1,000cc per minute and the charcoal adsorption pump should slowly cool to and remain below 5K.
5. The ^{3}He pot temperatures will slowly drop to 300mK followed closely by the sample temperature. The 1K pots should be monitored and adjusted as necessary to minimize their temperature and helium consumption.

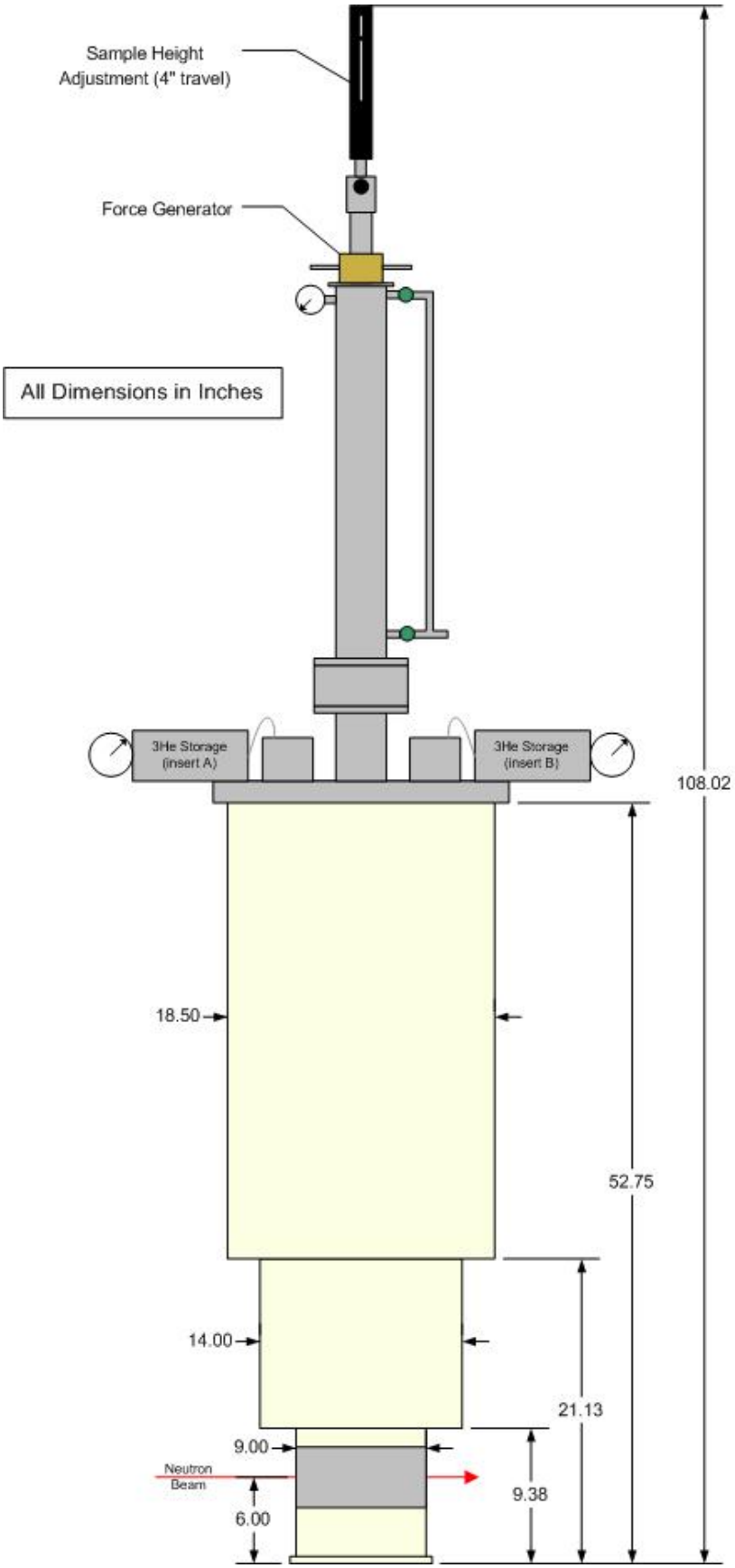
VII. Sample Change:

1. Connect a turbo pump to V-8 and evacuate the air lock assembly. Open V-7 and begin to slowly raise the sample stick. As the stick is pulled up it will frost up and can freeze the o-ring seal on the air lock. To avoid this pause every few minutes and allow the stick to warm or heat it with a heat gun. The clamp can be used to hold the stick in place as you are warming it up.
2. When the stick is all the way up and will not go any farther close V-9, stop the pump and unbolt the stick from the top of the cryostat. Helium gas can alternatively be added to the air lock to slowly warm the sample if necessary.
3. Cover V-9 with the blank flange to protect it.
4. To load another sample re-mount the stick and bolt it down. Evacuate the air lock with V-7 and V-8 open.
5. Have an experienced user add a small amount of ^{3}He gas to the IVC through V-10, close V-8, open V-9 and begin slowly lowering the stick. Continue pumping on V-7 to prevent any air from getting by the double o-ring seal there. The LHe will begin to rapidly boil off due to the large heat load. To decrease the helium boil off lock the stick in place and let it sit for fifteen minutes every few feet. The stick should be as cold as possible, but certainly less then 100K, before making contact with the mating surfaces in the sample well.
6. When the stick sensors reach approximately 25K pump out the IVC through V-10 using a turbo pump for at least a few hours.
7. Continue following the directions from step 5 of section V.

VIII. System Warm-up

1. Valves 2A/B and 5A/B should be closed to stop pumping on the 1K pots. Although there is cold He trapped between valve 2 and 5 there is an overpressure valve that will safely release any excess pressure.
2. Close valves 1A/B to seal the cooling circuit for the charcoal pumps and prevent air from being sucked in when the LHe runs out.
3. Finally loosen the force generator slightly so that it will be easier to remove at room temperature after the stick has expanded.
4. As long as the LHe reservoir is sealed as normal then the system should be safe to warm up on its own.

IV. Drawings


Sample Height
Adjustment (4" travel)
Force Generator
All Dimensions in Inches
3He Storage
(insert A)
3He Storage
(insert B)
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21.13
9.00
Neutron
Beam
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6.00